Surface Analysis of hydrogen loaded nickel alloys

E. Campari[*], S. Focardi[*], V. Gabbani[**], V. Montalbano[**], F. Piantelli[**], S. Veronesi[+**]

[*] Dipartimento di fisica, Università di Bologna – Centro I.M.O.

[**] Dipartimento di fisica, Università di Siena – Centro I.M.O.

[+] I.N.F.M. – UdR Siena

We present a surface analysis of nickel alloy rods loaded with hydrogen. By comparing these with a *blank* (unused) metal rod, morphological differences and a different composition of the surface are observed. These surface modifications follow a spatial distribution along the rod. These results are compared with a previous analysis of similar samples.

Introduction

In order to investigate the behavior of a metal loaded with hydrogen, we performed an accurate surface analysis on a nickel alloys sample loaded with hydrogen by mean of a Scanning Electron Microscopy (SEM). Previous experiments with these systems have produced very interesting phenomena such as excess heat production [1, 3, 7], alterations of elements on the metal surface [2, 7-9], neutron emission [4], and photon emission [5-7].

In this experiment a nickel alloy rod was loaded with hydrogen [5] and then analyzed. The same analysis was performed on a similar nickel alloy rod that had not been loaded with hydrogen. A comparison of the surface morphology and the elemental distribution shows many differences that seem to outline a spatial distribution of phenomena probably caused by the geometry of the experimental cell.

Hydrogen loading

The sample is a cylindrical rod made by a nickel alloy (NiCrFeMn 7.6-20.6-70.4-1.4) with length 9.0 cm. A chemical and a physical cleaning are performed before of sealing the sample in the experimental cell, showed in Fig. 1 and described in Ref. 3.

In the cell, annealing cycles are performed in vacuum and in a hydrogen atmosphere with the temperature in the range 400 to 700 K. The gas pressure is maintained in the range 100 to 1000 mbar. The hydrogen loading is described in Ref. 5.

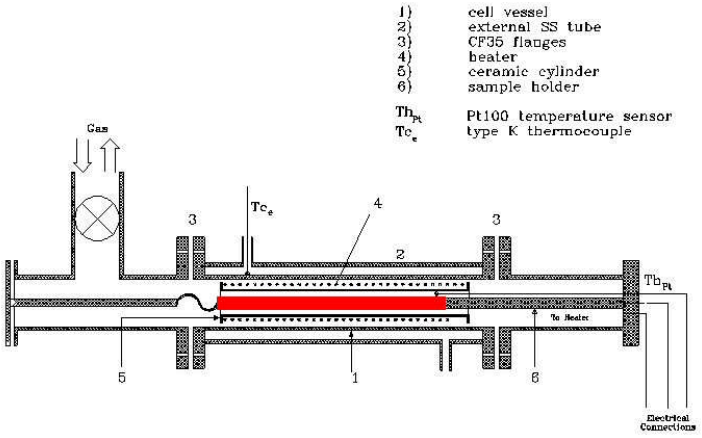


Figure 1. The experimental cell for hydrogen loading

Surface analysis

The surface of the sample has been analyzed by using a SEM which allows two different type of analysis: morphology and elemental distribution. An electron gun excites atoms in the surface (to a depth of few μm), secondary electrons (SE) emitted by the atoms and back-scattered electrons (BSE) allow us to obtain images of the surface.

Furthermore, an X–microprobe utilizing an energy-dispersive X–ray (EDX) system for elemental analysis, determines the elemental distribution on the surface in a quantitative fashion.

Morphological analysis

The *blank* rod shows uniform surface that always presents the same morphology along the cylindrical rod. In Fig. 2, a typical image of the surface is shown at different scales.

a) b) c)

Figure 2. *Blank* rod BSE. a) scale 200 μm, b) scale 50 μm, c) scale 10 μm.

In contrast, the surface of the used sample shows many differences along the cylindrical rod. For this reason a systematic investigation along the rod has been performed.

The surface near to the gas inflow is indistinguishable from the *blank* rod surface. Moving along the sample, the surface gradually changes up to the other end where the most impressive differences are observed. We have indicated the beginning of the rod where no changes are detected as $l = 0$.

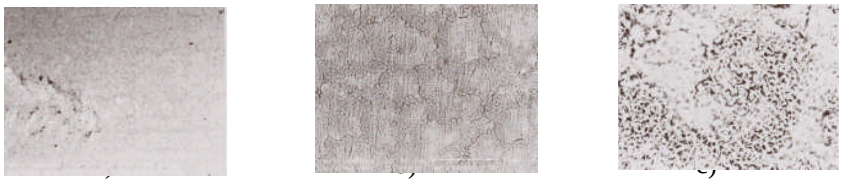
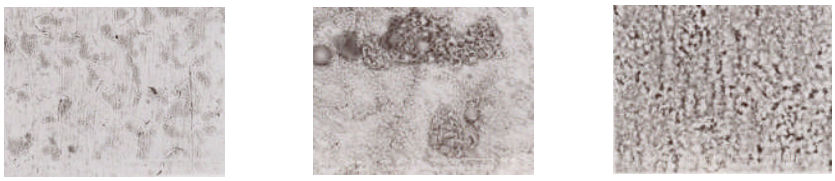


Figure 3. Sample rod at l = 2.0 cm BSE. a) scale 200 μm, b) scale 50 μm, c) scale 10 μm.

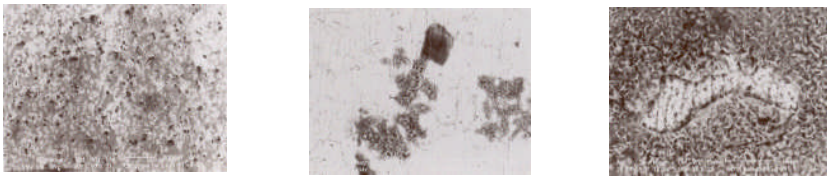
In Fig. 3, 4 and 5, the morphological alterations of the surface are shown. Initially, changes are distributed in isolated zones distributed uniformly.



a) b) c)

Figure 4. Sample rod at l = 5.0 cm BSE. a) scale 200 μm, b) scale 50 μm, c) scale 10 μm.

In particular, darker zones appear here and there; new structures similar to broken bubbles arise. The amount of altered surface increases along the rod. Completely altered surfaces are only found at the end of the rod. Only in one case, shown in Fig. 5c, it is still possible to recognize the initial surface in a region in which layers of the surface have been removed.



a) b) c)

Figure 5. Sample rod at l = 8.0 cm BSE. a) scale 200 μm, b) scale 50 μm, c) scale 10 μm.

In the images shown in this paragraph, the dimension of the spot and all other SEM parameters are optimized in order to obtain more details of the surface.

Elemental analysis

In order to quantitatively characterize the elemental distribution on the sample surface, all elemental analyses were performed under the same conditions, i.e. with the electron gun at 20 kV, spot dimension 2 - 6 nm, windows of 200 × 200 μm^2, with an acquisition time of 100 s. In these conditions, a *blank* rod analysis is shown in Fig. 6. The quantities of Ni, Cr, Fe and Mn remain the same along the rod, and surface analyses are indistinguishable from one region to another.

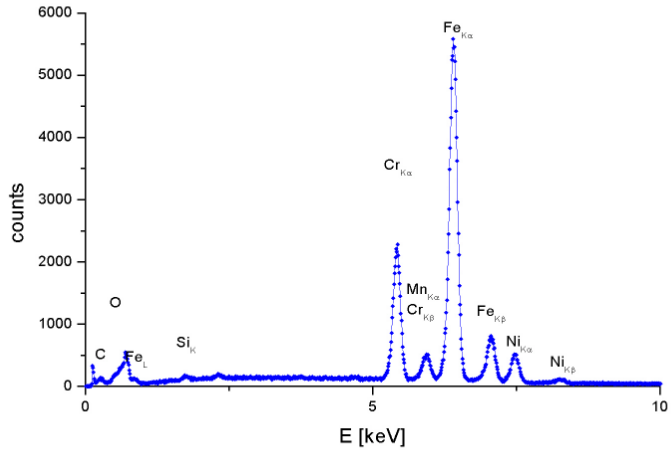


Figure 6. Elemental analysis of the *blank* rod

In contrast, the elemental analysis of the sample rod (Fig. 7) are very different from the *blank* rod analysis, and they are very different from one region to the next along the sample. This is why a systematic investigation along the sample was performed. We obtained the elemental distribution along the rod.

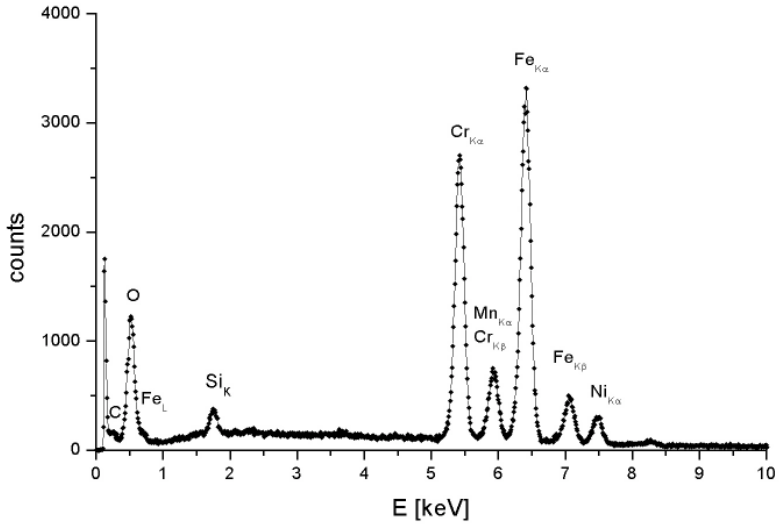


Figure 7. Example of elemental analysis of the sample

To correctly compare the components of the alloy, we note that the changes are essentially on the surface, i.e. at most in the first μm. This fact is confirmed by the elemental analysis performed in the central region showed in Fig. 5c, which is identical to an analysis of the *blank*. The X–microprobe interacts with atoms in few μm and for this reason we obtain information from atoms on the surface and in the bulk. A simple model

[2] allows us to separate the contribution to spectra of the element of the surface from the bulk and from other elements on the surface (for example O).

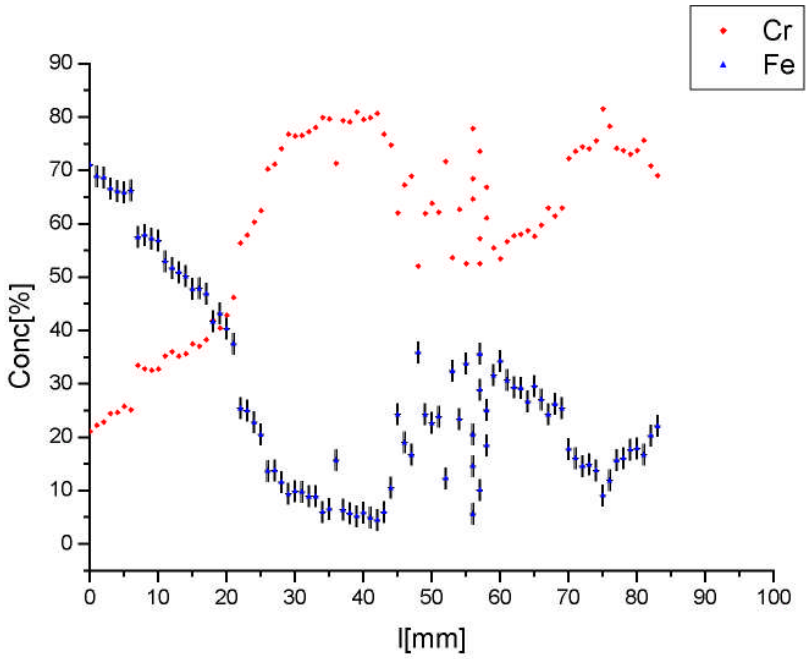


Figure 8. Spatial distribution of Cr and Fe in the sample. The measures on Fe show the bar error.

The most interesting results are shown in Figures 8, 9 and 10.

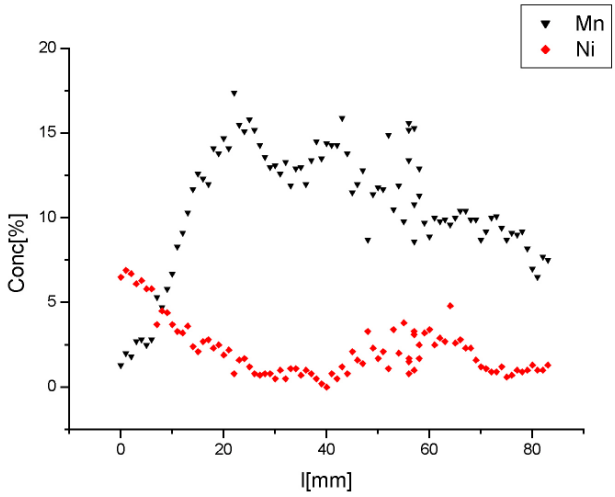


Figure 9. Spatial distribution of Ni and Mn in the sample

In Fig. 10, a quantity of Cu is measured in a narrow zone of the sample. Moreover, in a wide region of the sample, nickel is absent from the surface.

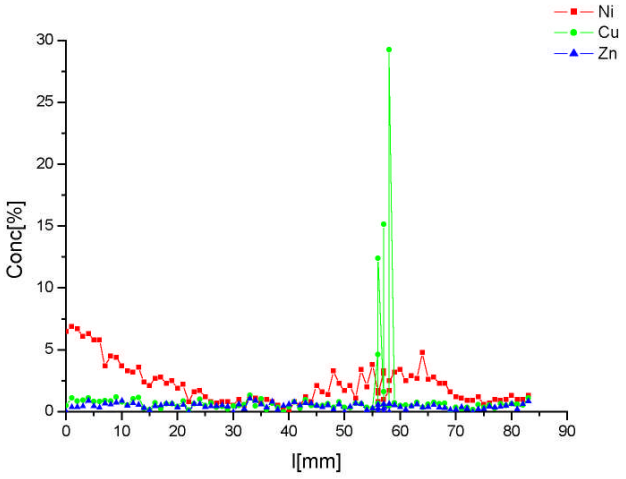


Figure 10. Spatial distribution of Ni and Cu on the sample surface.

Remarks and conclusion

The surface analysis of the nickel alloy confirm previous results [2, 9] obtained using a sample of the same composition in a similar experimental cell. In particular, the observed spatial distribution of the changes in the elements that appear on the surface seems to suggest an important contribution due to the geometry of the experimental cell. This geometry causes a temperature gradient and pressure that seems to drive the processes on the metal surface. Moreover, these processes are important and produce new elements on the surface without massive production of excess heat.

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